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Fracture Toughness and Stress Corrosion Resistance of U-0.75 wt.% Ti

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ARL-TR-750

April 1995

19950803 026

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REPORT DOCUMENTATION PAGE			Form Approved OMB No. 0704-0188	
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1. AGENCY USE ONLY (Leave blank)		2. REPORT DATE April 1995		3. REPORT TYPE AND DATES COVERED
4. TITLE AND SUBTITLE Fracture Toughness and Stress Corrosion Resistance of U-0.75 wt.% Ti			5. FUNDING NUMBERS	
6. AUTHOR(S) Chester V. Zabielski				
7. PERFORMING ORGANIZATION NAME(S) AND ADDRESS(ES) Army Research Laboratory Watertown, MA 02172-0001 ATTN: AMSRL-MA-CC			8. PERFORMING ORGANIZATION REPORT NUMBER ARL-TR-750	
9. SPONSORING/MONITORING AGENCY NAME(S) AND ADDRESS(ES)			10. SPONSORING/MONITORING AGENCY REPORT NUMBER	
11. SUPPLEMENTARY NOTES				
12a. DISTRIBUTION/AVAILABILITY STATEMENT Approved for public release; distribution unlimited.			12b. DISTRIBUTION CODE	
13. ABSTRACT (Maximum 200 words) See Reverse				
14. SUBJECT TERMS Uranium Alloy, Fracture Toughness, Stress Corrosion, Heat Treatment, Hydrogen Content			15. NUMBER OF PAGES 35	
			16. PRICE CODE	
17. SECURITY CLASSIFICATION OF REPORT Unclassified	18. SECURITY CLASSIFICATION OF THIS PAGE Unclassified	19. SECURITY CLASSIFICATION OF ABSTRACT Unclassified	20. LIMITATION OF ABSTRACT UL	

Abstract

As a consequence of the several failures of U-0.75 wt% Ti circumferentially grooved machined bars stressed at lower than normal ambient temperatures (-37°C to -46°C), an intensive fracture toughness study of the U-0.75 wt% Ti material was carried out. The objective of the study was to determine the effect of heat treatment, microstructure, hardness, mechanical properties and test temperature on fracture toughness. Fracture toughness measurements in the temperature range of -73°C to $+38^{\circ}\text{C}$ were made for the alloy processed by (1) alpha extrusion, gamma vacuum solution treatment, directionally quenching in H_2O , aging; (2) gamma rolling, gamma solution treatment in molten salt, plunge quenching in oil, aging; (3) solution treatment in vacuum for the material processed in (2), directionally quenching in H_2O and re-aging. Two types of fracture toughness specimens were considered. Based on preliminary test data, a slow-bend precracked Charpy specimen was selected for final measurements. Data obtained was compared with the meager fracture toughness data for the alloy reported in the literature. Based on these data, a minimum fracture toughness requirement at -46°C was recommended. K_{Isc} measurements were also made and the data compared with previously reported results.

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Introduction

An investigation was conducted of the several failures of U-0.75 wt% Ti circumferentially grooved machined cylindrical bars which were stressed at lower than normal ambient temperatures (-39°C to -46°C). A simple fracture mechanics approach suggested that poor low temperature fracture toughness of the alloy was contributory.

As a consequence, a systematic investigation of the fracture toughness of the U-0.75 wt% Ti machined bars was carried out. The U-0.75 wt% Ti alloy was provided by two sources N and B. The failed machined bars were processed by Source N. Another lot of U-0.75 wt% Ti alloy was also obtained from Source R for comparison. Representative machined bars from each source were fully characterized and processing parameters, mechanical properties, and microstructure were correlated with fracture toughness as a function of test temperature.

Materials

The Source N machined bars were fabricated from a 35.6 mm diameter rod which was rolled from 203.2 mm diameter ingots. The bars were solution treated for 10 minutes at 899°C in molten salt, plunge oil quenched, and aged at 350°C in a lead bath.

Six bars, 152.4 mm long and 35.6 mm in diameter, were received from Source B. These bars were the bottom portions of longer 406.4 mm bars and the first to enter the water on vertical quench. The 406.4 mm long extruded bars were vacuum solution treated at 800°C for two hours and at 850°C for one-half hour, vertically water quenched at 0.46 m per minute, and aged at 350°C in a lead bath for 16 hours.

The Source R machined bars were fabricated from 35.6 mm diameter bars which were alpha extruded from 101.6 mm diameter ingots. The ingots were homogenized in vacuum at 1050°C for six hours prior to extrusion. The extruded

bars were then solution treated for two hours at 800°C and one-half hour at 850°C, vertically water quenched at 0.46 m per minute, and aged at 350°C in a lead bath for 16 hours.

Four additional 35.6 mm diameter bars which were received from Source N in the as-rolled condition were given STA treatments comparable to Source B and R processing; i.e., they were vacuum solution treated for two hours at 800°C and one-half hour at 850°C, vertically quenched in water at 0.53 m per minute, and aged in vacuum at 350°C, 370°C, or 390°C, respectively, for seven hours.

Fracture Toughness Test Procedures

Sampling

Two types of fracture toughness specimens were utilized: (a) a single edge-notched bend specimen conforming to plane strain requirements (K_{IC}) of ASTM E 399-74 (FT1), and (b) a slow-bend V-notched Charpy impact specimen (CV2) for approximate K_{IC} or K_Q . Both types of specimens were used for **static** fracture toughness measurements. The Charpy-type specimen was also used for **dynamic** fracture toughness K_{ID} . Regardless of the type of specimen, the notches were always machined from the outer diameter of the bar so that the microstructure in the vicinity of the notch would be comparable to that of the groove in the original application.

From each of four machined bars representative of Source N lots which failed at low temperature, two Charpy, K_Q specimens and two K_{IC} specimens were cut alternately starting at the nose; i.e., the end which entered the water first during the vertical quench. A total of four Charpy and four K_{IC} specimens were cut per machined bar. In a similar fashion, four K_{IC} specimens and four K_Q specimens were machined from three Source R machined bars. Based on the similarity of K_{IC} , K_Q , and K_{ID} values obtained, it was decided to concentrate on the simplest

and least costly specimen, the V-notch bend Charpy impact specimen only, and report K_Q values for the remaining materials evaluated. Therefore, only K_Q specimens were machined from four Source N as-rolled bars which had been vacuum solution treated, vertically water quenched, and aged, and from six Source B bars which were similarly heat treated. In addition, tension and K_{Isc} specimens were fabricated from the above materials to confirm the specified strength requirement and to determine susceptibility to stress corrosion cracking.

The stress corrosion specimens which were single edge notch specimens (76.2 x 5.08 x 5.08 mm) were cut with the long dimension parallel to the direction of maximum grain flow and notched so that crack growth and fracture would occur in the radial direction (see Figure 1).

Test Method

The procedure for K_{IC} measurement involved three-point bend testing of notched specimens that had been precracked in fatigue. Load versus displacement across the notch was recorded autographically. The K_{IC} value was calculated from the load corresponding to a 2% increment of crack extension by equations which have been established on the basis of elastic stress analysis of bend specimens. The detailed procedure is described in ASTM E 399-74. The method for K_Q measurement employed a Charpy specimen provided with a sharp notch terminating in a fatigue crack tested in three-point bending. The maximum load in the test was recorded and the nominal crack length was determined from this value, as well as the original dimensions of the specimen using the single beam equation. A detailed description is contained in the proposed ASTM E24.03.03 draft dated February 7, 1979. Precracking of specimens for both test procedures involved initiation of the crack and subsequent growth in tension. The dynamic fracture toughness, K_{ID} , was measured using an impact test machine with an instrumented

Charpy tup. The hammer of the testing equipment had a velocity of 4 ft/sec at impact. Load and energy as a function of time were recorded during each test. The fracture load was used to calculate K_{ID} values using the equation for three point bend specimens according to ASTM E 399-78. The Rockwell C hardness of each specimen was measured by taking the average of four equally spaced readings on the back of each specimen.

The method for stress corrosion measurements follows. The test uses a precracked bar stressed as a cantilever beam. A sharp notch machined across the rectangular bar specimens at mid-length is sharpened by fatiguing. The specimen is held in a rack horizontally with the precracked central portion surrounded by a plastic bottle which contains the environment. One end of the specimen is clamped to the mast of the rack and the other end to an arm from which weights are suspended. On evaluating the alloy, one specimen is first stressed in air at increasing loads until it fractures. The data are reduced to stress intensity using the Kies equation (see Figure 1). Having established stress intensity for dry conditions (K_{IC}), other specimens are similarly tested in distilled H_2O and NaCl solutions at a somewhat lower stress intensity. If the specimen did not fail within an hour, the stress intensity was increased by approximately 3% each succeeding hour until failure occurred and the time required for rupture noted. Additional specimens were stressed at decreasingly lower stress intensities for 1000 hours or until failure occurred to give a more valid value for K_{Isc} . K_{Isc} is the threshold stress intensity value for the onset of cracking which was determined from a plot of stress intensity versus time to failure.

Results

Comparison of Failed Source N Versus the Source R Processed Material

Chemistry, Microstructure, Mechanical Properties

Table 1 summarizes mechanical properties and chemistries for Source N and R machined bars. Major differences were observed in hydrogen content, elongation, and RA values. The Source N material exhibited higher H and lower elongation and RA.

The structure of the Source N machined bars is shown in Figure 2. The view is perpendicular to the extrusion direction at the diameter and represents slightly more than one-half of the complete cross section. A coarse duplex grain size is observed along with banding and centerline porosity or voids.

The microstructure of a Source R machined bar is shown at both the nose section (where the bar entered the water first on vertical quenching), as shown in Figure 3, and at the tail, or rear portion of the bar which entered the water last, as shown in Figure 4. The microstructure in Figure 3 is essentially martensitic with evidence of incipient slack quench at the grain boundaries; small voids, particularly in the central area are observed. The tail, or rear, views show a more pronounced slack quench and even larger voids, particularly in the central areas, as shown in Figure 4.

Fracture Toughness Versus Temperature

Figure 5 compares fracture toughness data for the failed Source N machined bar material obtained from the two types of specimens employed. The data was designated K_{IC} if all the conditions of ASTM E 399-74 were met; otherwise, the values were designated K_Q .

All K_{IC} and K_Q values were below $33 \text{ MPa}\sqrt{\text{m}}$ the recommended standard above which the bars are found not to fail regardless of test temperature. For the vacuum solution treated, vertically water quenched and aged bars all fracture toughness values were greater than 33 MPa for test temperatures above -73°C . The K_{IC} and K_Q values were in fair agreement. The average value at -46°C was $24 \text{ MPa}\sqrt{\text{m}}$, and at 24°C , $30 \text{ MPa}\sqrt{\text{m}}$.

Previous work has shown that fracture toughness values for titanium and steel alloys obtained with compact tension and bend specimens conforming to ASTM E 399-74, were in good agreement with those obtained with precracked Charpy specimens up to values of $44 \text{ MPa}\sqrt{\text{m}}$ [1,2].

Fracture Toughness Versus Hardness

Figure 6 shows a plot of fracture toughness versus HRC hardness values for individual specimens taken from the failed Source N machined bar lots and the Source R machined bars. The slightly softer vacuum solution treated and vertically water quenched Source R machined bars had significantly higher fracture toughness values than the Source N machined bar lots which were molten salt solution treated, plunge quenched in oil, and had higher hydrogen. At both 24°C and -46°C , fracture toughness values for specimens from the Source R machined bar lots were greater than $35 \text{ MPa}\sqrt{\text{m}}$. All values were below $33 \text{ MPa}\sqrt{\text{m}}$ for specimens from the Source N machined bar lots.

Dynamic Fracture Toughness Versus Hardness

Figure 7 shows a plot of dynamic fracture toughness K_{ID} versus HRC hardness values for individual specimens of the failed Source N machined bar lots and the Source R machined bar lots. The slightly softer Source R machined bar lots had significantly higher dynamic fracture toughness values than those of the

Source N machined bar lots. All dynamic fracture toughness values of specimens from Source R machined bar lots were greater than $38 \text{ MPa}\sqrt{\text{m}}$. Source N machined bar lot values were below $33 \text{ MPa}\sqrt{\text{m}}$. The K_{ID} data were in good agreement with the K_{IC} and K_Q values.

Source N As-Rolled Bars Vacuum Solution Treated, Vertically Water Quenched and Aged

Chemistry, Mechanical Properties

The chemical composition of the as-rolled Source N bars is shown in Table 2. All chemical properties except hydrogen meet the requirements of the standard. The 1.8 ppm hydrogen exceeds the maximum requirement of 1 ppm. Table 3 summarizes mechanical properties for the alloy aged at three different temperatures: 350°C , 370°C , and 390°C . In all three cases, the mechanical properties meet or exceed the minimum requirements specified for the heat treated Source N U-0.75 wt% Ti bars. Data from the unaged material is included for comparison.

Fracture Toughness Versus Temperature

Fracture toughness (K_Q) of the above mentioned materials were determined utilizing precracked Charpy specimens at test temperatures ranging from -73°C to 21°C (RT). The data are recorded in Table 4 and plotted in Figure 8. It should be noted that a limited number of specimens were available for test. Generally, fracture toughness increased with test temperature. The unaged alloy (solution treated and quenched) gave the highest fracture toughness values. As the aging temperature increased, fracture toughness decreased. The bars aged at 390°C gave the lowest fracture toughness values. Fracture toughness (K_Q) values were greater than $38 \text{ MPa}\sqrt{\text{m}}$ for all aged bars at the -46°C and higher test temperatures. These data show that the fracture toughness of the Source N material can be substantially improved by changing the heat treatment procedure from solution treatment in molten

salt and fully plunge quenching in oil to solution treatment in vacuum and vertically quenching in water.

Fracture Toughness Versus Hardness

Figure 9 plots fracture toughness (K_{Ic} MPa \sqrt{m}) versus HRC hardness for the unaged and aged bars. Room temperature fracture toughness values decreased significantly with increase in HRC hardness and aging temperature. At the -46°C and -73°C test temperatures the rate of decrease of fracture toughness values with increase in HRC hardness and aging temperature decreased markedly. Above -46°C with increasing test temperatures the values of fracture toughness rose significantly with the greatest rise occurring for the unaged and the 350°C aged samples with the lowest for 390°C aged samples. The rapid increase in the slopes (the rate of increase of fracture toughness values with decrease in HRC hardness) above -46°C, indicate that -46°C is the nominal transition temperature above which fracture toughness improves more rapidly. This transition temperature is also readily observed from the increase in the slope above -46°C in Figure 12 for the U-0.75 wt% Ti Source B STA alloy. The steepest slope occurred at the room test temperature with the most improvement in fracture toughness occurring for the lower hardness samples in the unaged and 350°C aged conditions.

Source B Bars Vacuum Solution Treated, Vertically Water Quenched and Aged at 350°C for 16 Hours

Chemistry, Mechanical Properties

Table 5 shows the chemical properties for the Source B processed alloy. Note that the hydrogen content is 0.5 ppm.

The bars were heat treated to a narrow hardness range (39 HRC to 40 HRC) as illustrated in the histogram for a typical bar (see Figure 10).

Figure 11 summarizes HRC traverse data taken across the diameter of transverse sections for six bars at 45° angles at the vertically water quenched end, marked A (first hits H₂O), and 152 mm from the end, marked B. The bars at position B were slightly harder than at position A. The central areas of the bars were quite uniform in hardness and slightly softer.

The tensile properties of the six aged U-0.75 wt% Ti bars versus temperature are shown in Table 6. The yield strength (YS) was found to increase slightly with decrease in test temperature. The 0.2% yield strength of the material exceeds the minimum requirement of 724 MPa of the standard.

Fracture Toughness Versus Test Temperature

Figure 12 plots fracture toughness versus test temperature from -73°C to 38°C. Four test values were obtained at each temperature and lines were drawn through the outermost points to show the band of values. Fracture toughness increased with increasing test temperature. There was no evidence of change or decrease in slope at the 38°C test temperature, but below -46°C the slope decreased indicating a less ductile region. The average K_Q value for each test temperature is shown in Table 7. Note that the average K_Q value at -46°C is 40 MPa√m., which exceeds the established minimum requirement of 33 MPa√m.

Stress Corrosion Cracking

Table 8 compares the critical stress intensity for crack propagation in an aqueous solution containing 50 ppm Cl-(K_{Isc}) for 1000 hours of the Source N processed U-0.75 wt% Ti alloy (solution treated in molten salt and plunge quenched in

oil and aged) with the Source R processed alloy (vacuum solution treated and vertically water quenched and aged). The Source R U-0.75 wt% Ti alloy is less susceptible to stress corrosion than the Source N material due to the differences in processing. Crack extension in all of the alloys was transgranular and failure occurred by brittle quasicleavage fracture in NaCl solution [3,4].

Ratio Analysis Diagrams (RAD)

$$K_{IC}/\sigma_{YS}$$

The best index of a material's fracture resistance is the K_{IC}/σ_{YS} ratio since it is this ratio of materials properties that determines flaw size and applied stress which are the parameters of interest to designers. The so-called ratio analysis diagram (RAD) [5,6] encompasses the range of strength and fracture resistance. Its framework is formed from the scales of YS versus K_Q . The technological limit line represents the highest values of fracture resistance measured to date.

Figure 13 contains the RAD constructed for the U-0.75 wt% Ti alloy [7,8]. The envelope "B" encompasses fracture toughness data obtained for the Source N processed alloy which are representative of the failed low temperature machined bar lots. This material was molten salt solution treated, quenched in oil, and aged; it also contained high hydrogen (>1 ppm). Envelopes "A" and "D" contain data for Source R and N bars, respectively, which were vacuum solution treated, vertically water quenched and aged and had a low hydrogen content (<1 ppm). Envelope "C" includes data for bars with hydrogen contents >1 ppm and with incompletely martensitic structures.

The data shows that the fracture toughness of the alloy is highly sensitive to variations in heat treatment and concomitant interstitial content and microstructure. Under optimum conditions a fracture toughness of $88 \text{ MPa}\sqrt{\text{m}}$ has been

reported for the U-0.75 wt% Ti alloy at a YS of 793 MPa. Further processing improvements and alloy development may raise this current limit to $99 \text{ MPa}\sqrt{\text{m}}$.

K_{Isc}

The RAD shown in Figure 13 superimposes K_{Isc} data on the fracture toughness data displayed in Figure 14. The envelope shown contains earlier K_{Isc} data obtained in 50 ppm Cl⁻ solution and represents different sources of material, laboratories, and processing procedures. This material includes data for bars with hydrogen contents >1 ppm and with incompletely martensitic and nonmartensitic microstructure. The data reported in Table 8 are shown above the envelope and the highest K_{Isc} of $25 \text{ MPa}\sqrt{\text{m}}$ which is in good agreement with other published data [9,10] represents a critical flaw size of 0.2 mm for crack propagation in the chloride solution. The other data represent tolerance to even smaller critical flaw sizes.

Conclusions

It was shown that the fracture toughness of the U-0.75 wt% Ti alloy is highly sensitive to variations in heat treatment and concomitant interstitial content and microstructure. The Source N processed U-0.75 wt% Ti alloy representative of the failed machined bars (low temperature) had appreciably lower fracture toughness ($22 \text{ MPa}\sqrt{\text{m}}$ at -46°C) than the alloy processed either by Source B or R ($35 \text{ MPa}\sqrt{\text{m}}$ at -46°C).

The failed Source N material was characterized as high hydrogen content (2 to 4 ppm), low elongation (7%) material with microstructural features that included a coarse grain size, duplex structure, banding, and centerline porosity.

By comparison, the Source B and Source R processed alloy contained less hydrogen (<1 ppm), exhibited higher elongation (14%), a brittle to ductile transition.

and essentially a martensitic structure with small voids in the central area. However, it was demonstrated that the Source N material could achieve comparability of fracture toughness to the Source B and Source R processed alloy by solution treatment in vacuum, vertically water quenching, and aging instead of solution treatment in molten salt, fully plunge quenching in oil, and aging. Based on the extensive fracture toughness testing of these bars similarly processed, a minimum fracture toughness requirement of $33 \text{ MPa}\sqrt{\text{m}}$ at -46°C was established.

The U-0.75 wt% Ti alloy is very susceptible to stress corrosion cracking in aqueous chloride solutions (K_{Iscc} 20 to $25 \text{ MPa}\sqrt{\text{m}}$). There is an improvement in resistance to stress corrosion for the low hydrogen (<1 ppm) essentially martensitic bars which have been vacuum solution treated, water quenched, and aged.

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Table 1. Comparison of Source R and N machined bars

Property/Chemistry	Source R	Source N
Ultimate (MPa)	1448	1351
Yield (MPa)	793	786
Elongation (%)	12 - 16	5 - 9
RA (%)	12 - 16	4 - 8
Hardness (HRC)	38 - 43	40 - 42
Ti (%)	0.69 - 0.73	0.69 - 0.71
C (ppm)	<100	<40
H (ppm)	<1	2 - 4

Table 2. Chemical analysis data for as-received Source N bars

Ti	0.72%	Mn	8 ppm
C	19 ppm	Cu	7 ppm
H	1.8 ppm	Mg	< 4 ppm
Si	60 ppm	Ba	< 3 ppm
Fe	34 ppm	Cr	2 ppm
Al	14 ppm	Be	< 1 ppm
Ni	10 ppm	B	< 1 ppm
Pb	9 ppm	Sn	< 1 ppm
Zn	<20 ppm	V	< 1 ppm
Density = 18.64			

Table 3. Mechanical properties of aged U-0.75 wt% Ti Source N bars

	Hardness (HRC)	YS (0.2%)* (MPa)	TS (MPa)	Elong* (%)	RA* (%)
Unaged	36.2	641	1292	17.9	16.5
Aged for 7 Hours at					
350°C	37.5	745	1324	17.2	17.4
370°C	39.0	752	1351	13.9	18.7
390°C	41.5	797	1472	12.5	14.9

All bars solution treated at 800°C for two hours, 850°C for one-half hour and vertically water quenched at 0.53 m per minute

*Average of 4 values

Table 4. Fracture toughness (K_{Ic}) of aged U-0.75 wt% Ti Source N bars

	Test Temperature (°C)				
	-73	-46	-29	-7	R.T.*
	K_{Ic} (MPa \sqrt{m})				
Unaged	37	42	51	59	68
Aged for 7 hours at					
350°C	38	40	45	53	60
370°C	32	39	48	47	64
390°C	32	39	43	47	47

All bars solution treated at 800°C for two hours and 850°C for one-half hour and vertically water quenched at 0.53 mm per minute

*Average of 2 values

Table 5. Chemical analysis of Source B bars (101, 103, 104, 105, 107, 108) from 114.3 mm diameter ingot

Ingot Analysis	
Ti Center	0.73%
Ti Bottom	0.73%
H	0.5 ppm
C	70-80 ppm
Al	5 ppm
Si	45 ppm
Fe	30 ppm
Nb	<10 ppm
Ni	25 ppm

Table 6. Variation of tensile properties of aged U-0.75 wt% Ti Source B* bars with temperature

Temp (°C)	YS 0.1% (MPa)	YS 0.2% (MPa)	ULT (MPa)	E (GPa)
21	696	786	1372	141
4	717	800	1351	133
-7	703	793	1420	128
-29	731	827	1448	134
-46	758	855	1420	134
-73	745	841	1379	141

*Source B 35.6 mm diameter U-0.75 wt% Ti bars 101-108 solution treated at 800°C for two hours, and 850°C for one-half hour; vertically water quenched at 0.45 m per minute; aged for 16 hours at 350°C lead bath (114 mm diameter ingot α extruded).

NOTE: Averages of 2 values

Table 7. Variation of fracture toughness of aged U-0.75 wt% Ti Source B bars with temperature

Temperature (°C)	Hardness* (HRC)	K _Q † (MPa √m)
38	39.4	74
21	39.5	65
4	39.7	61
-7	39.7	52
-20	39.6	50
-29	39.4	46
-46	39.4	40
-73	39.7	35

*Average of 16 values taken at RT

†Average of 4 values

Source B 35.6 mm diameter U-0.75% Ti bars 101-108 solution treated at 800°C for two hours and 850°C for one-half hour, vertically water quenched at .46 m per minute and aged 16 hours at 350°C in lead bath (114 mm in diameter ingot α extruded).

Table 8. K_{ISCC} data for Source N and R machined bars in 50 ppm Cl-

Sample	No.	K _{ISCC} MPa √m
Source N	8	20
Source R	6	25

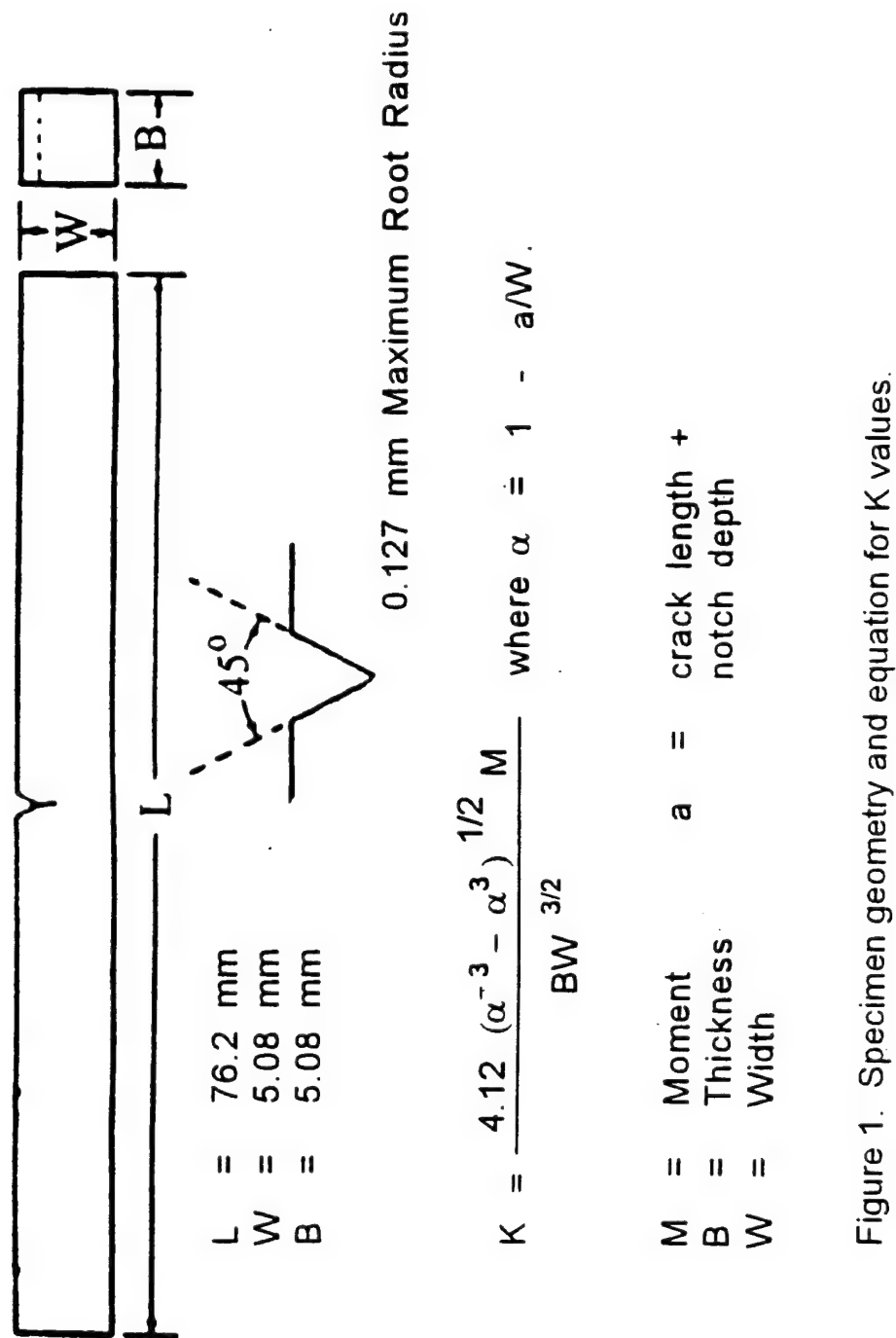


Figure 1. Specimen geometry and equation for K values.

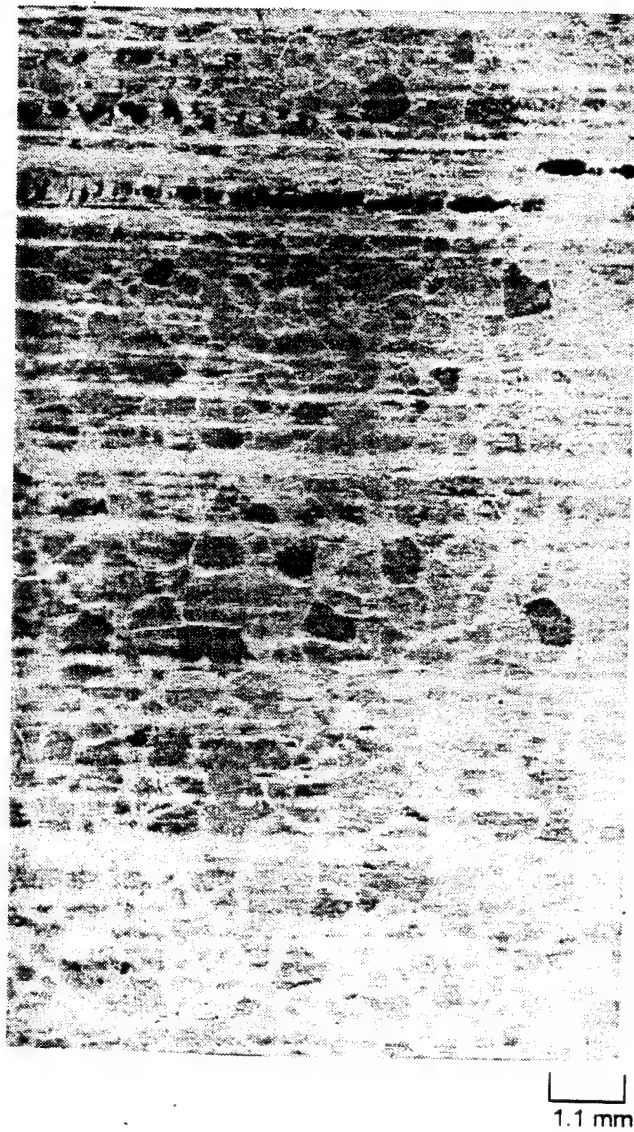
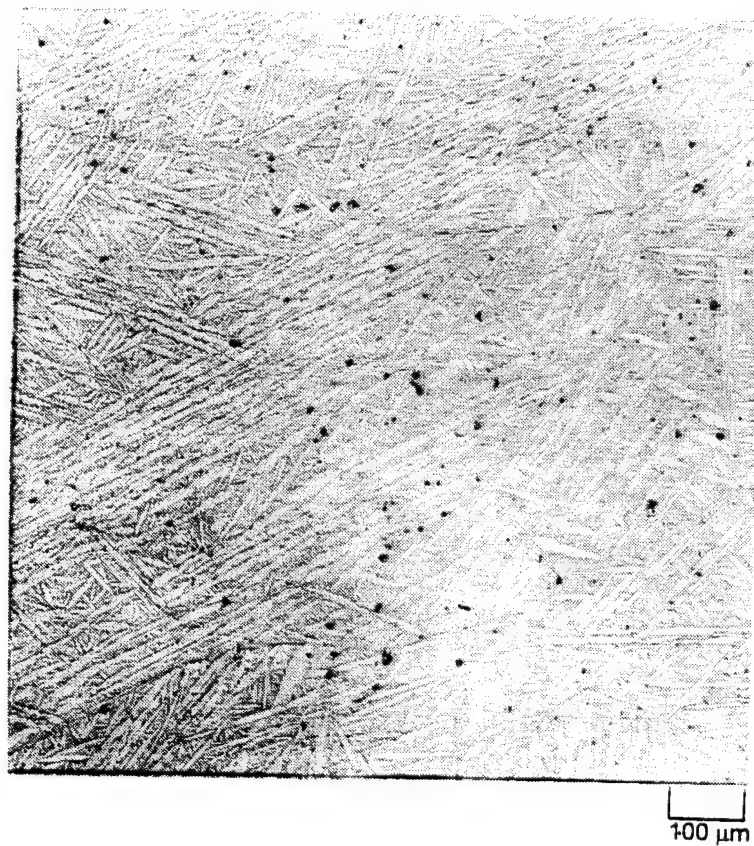
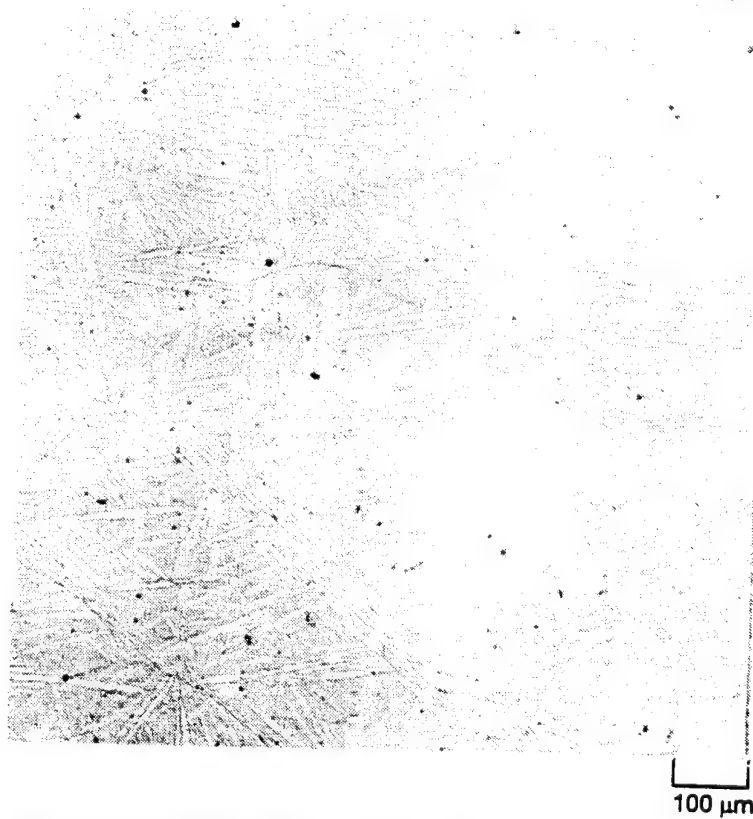


Figure 2. U-0.75 wt% Ti (Source N) - solution treated (molten salt) 899°C for 10 minutes, oil quenched, and aged at 350°C for one hour.

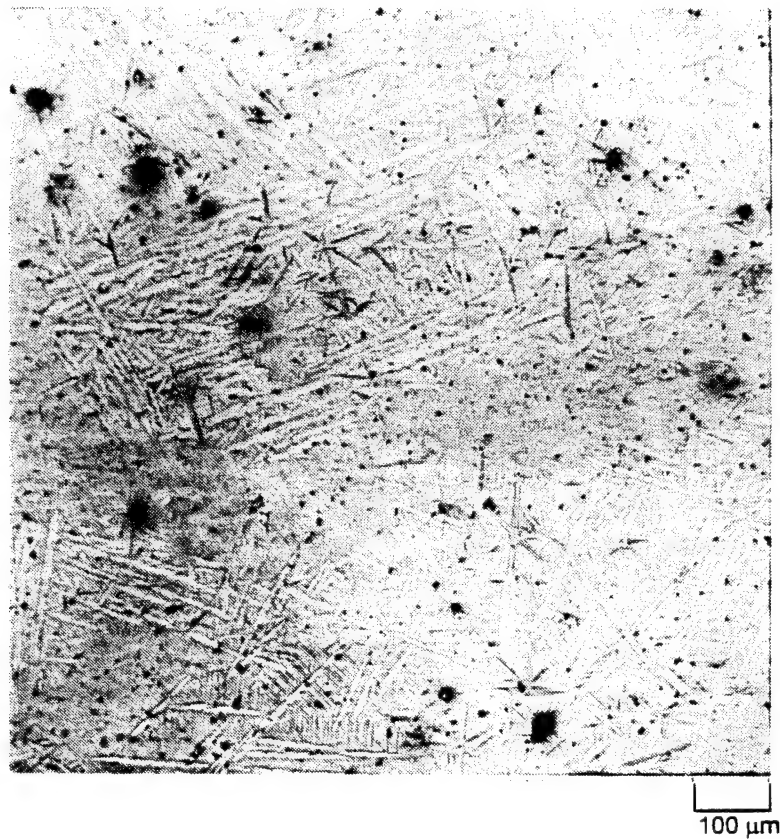


Center

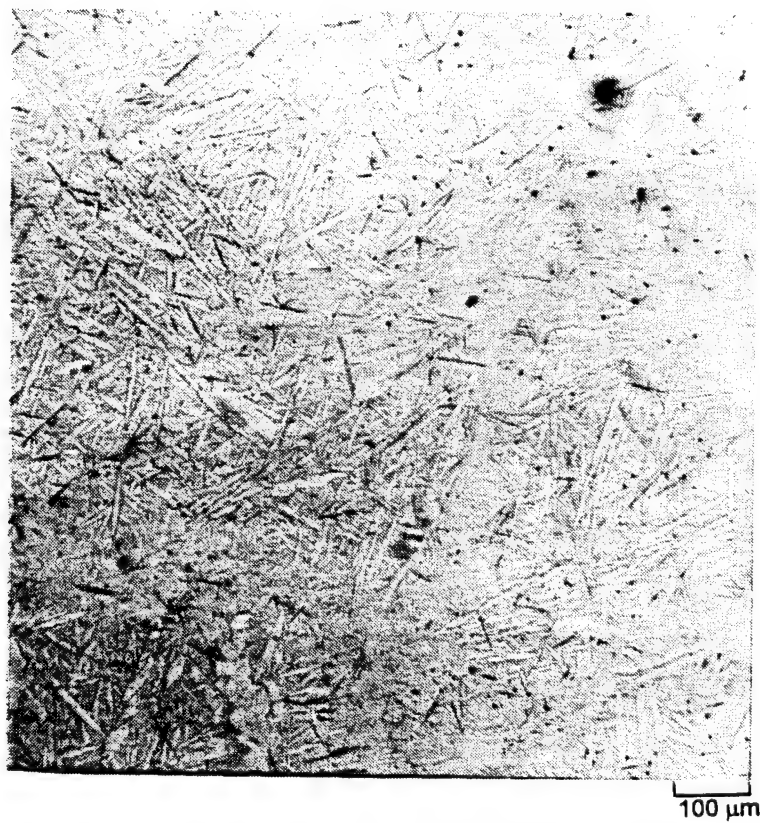


Edge

Figure 3. U-0.75 wt% Ti (Source R) lower nose section - solution treated at 800°C for two hours; 850°C for one-half hour; vertically water quenched 0.46 m per minute; aged (lead bath) for 16 hours at 350°C.



Center



Edge

Figure 4. U-0.75 wt% Ti (Source R) upper tail section - solution treated at 800°C for two hours; 850°C for one-half hour, vertically water quenched 0.46 m per minute; aged (lead bath) for 16 hours at 350°C.

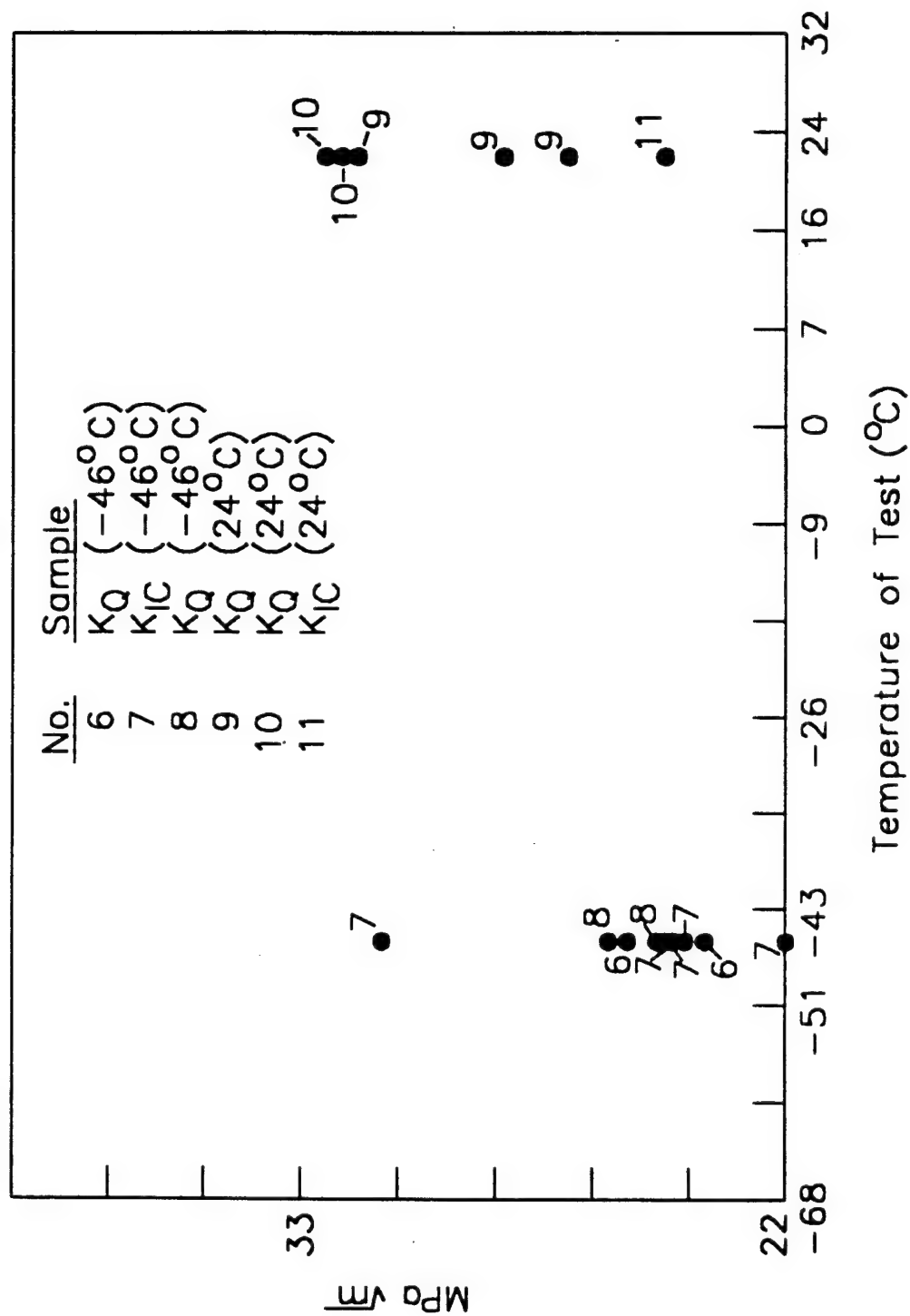


Figure 5. Fracture toughness of aged U-0.75 wt% Ti Source N machined Bars versus temperature of test.

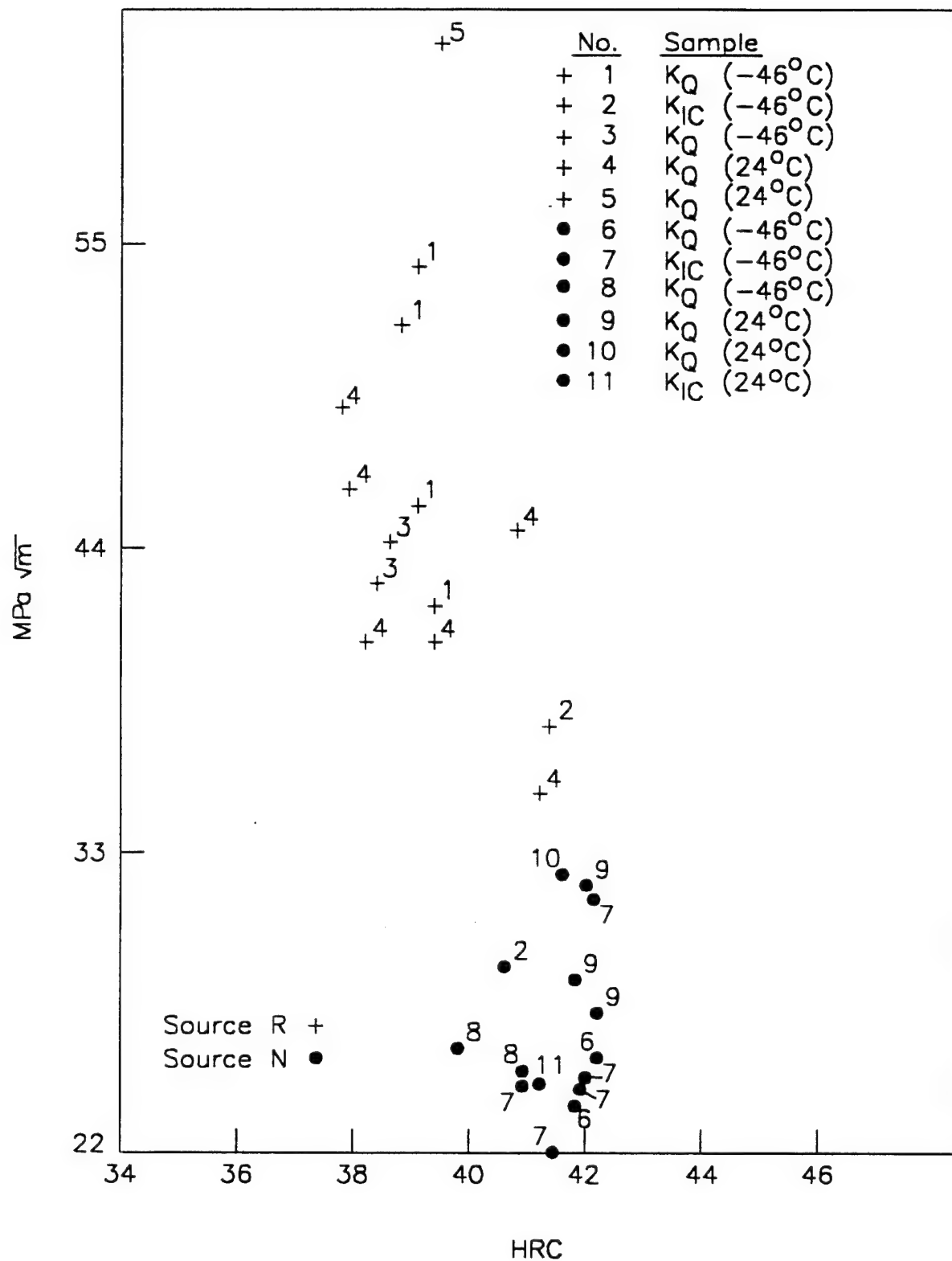


Figure 6. Fracture toughness of aged U-0.75 wt% Ti Source R and N machined bars versus HRC.

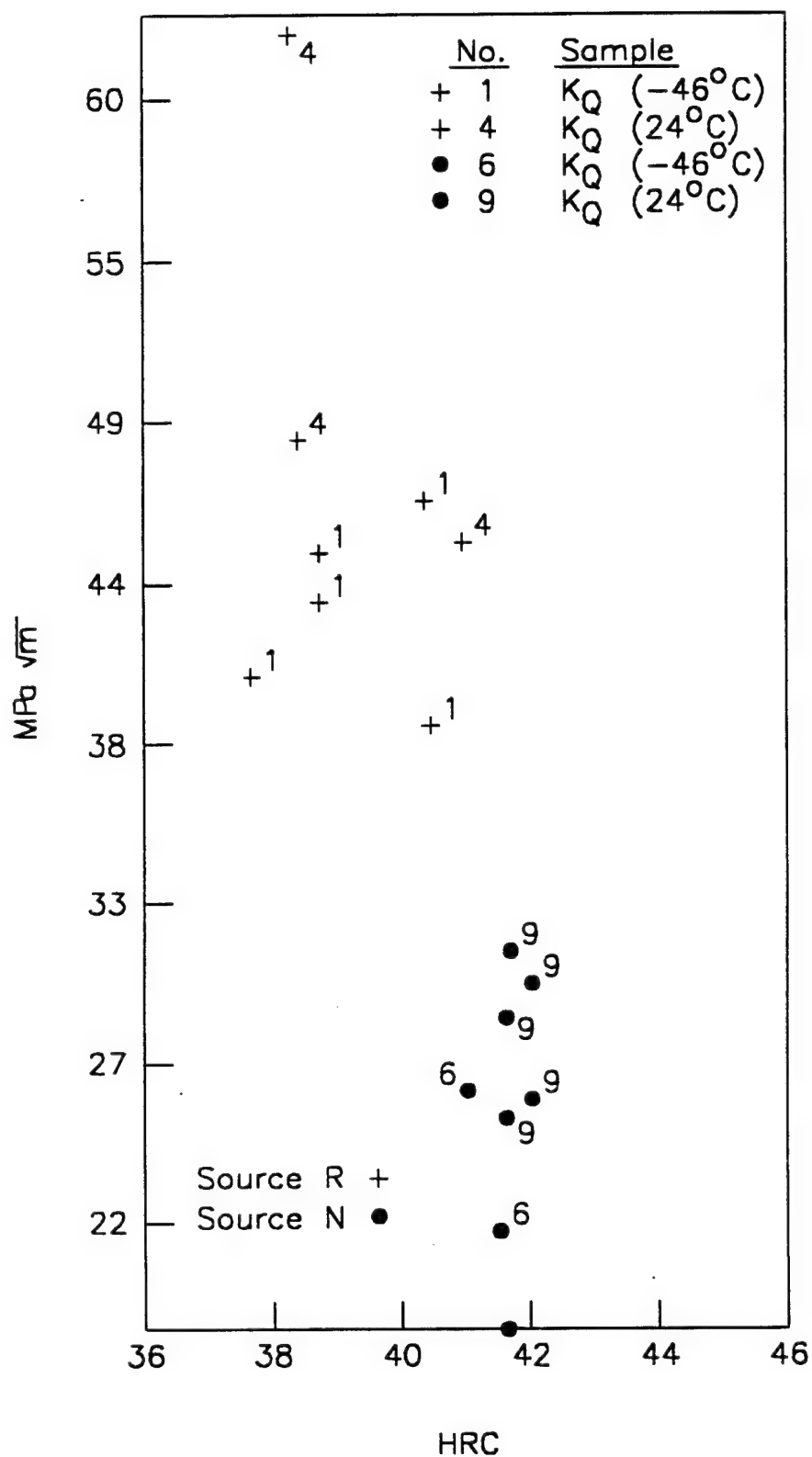


Figure 7. Dynamic fracture toughness of aged U-0.75 wt%Ti Source R and N machined bars versus HRC.

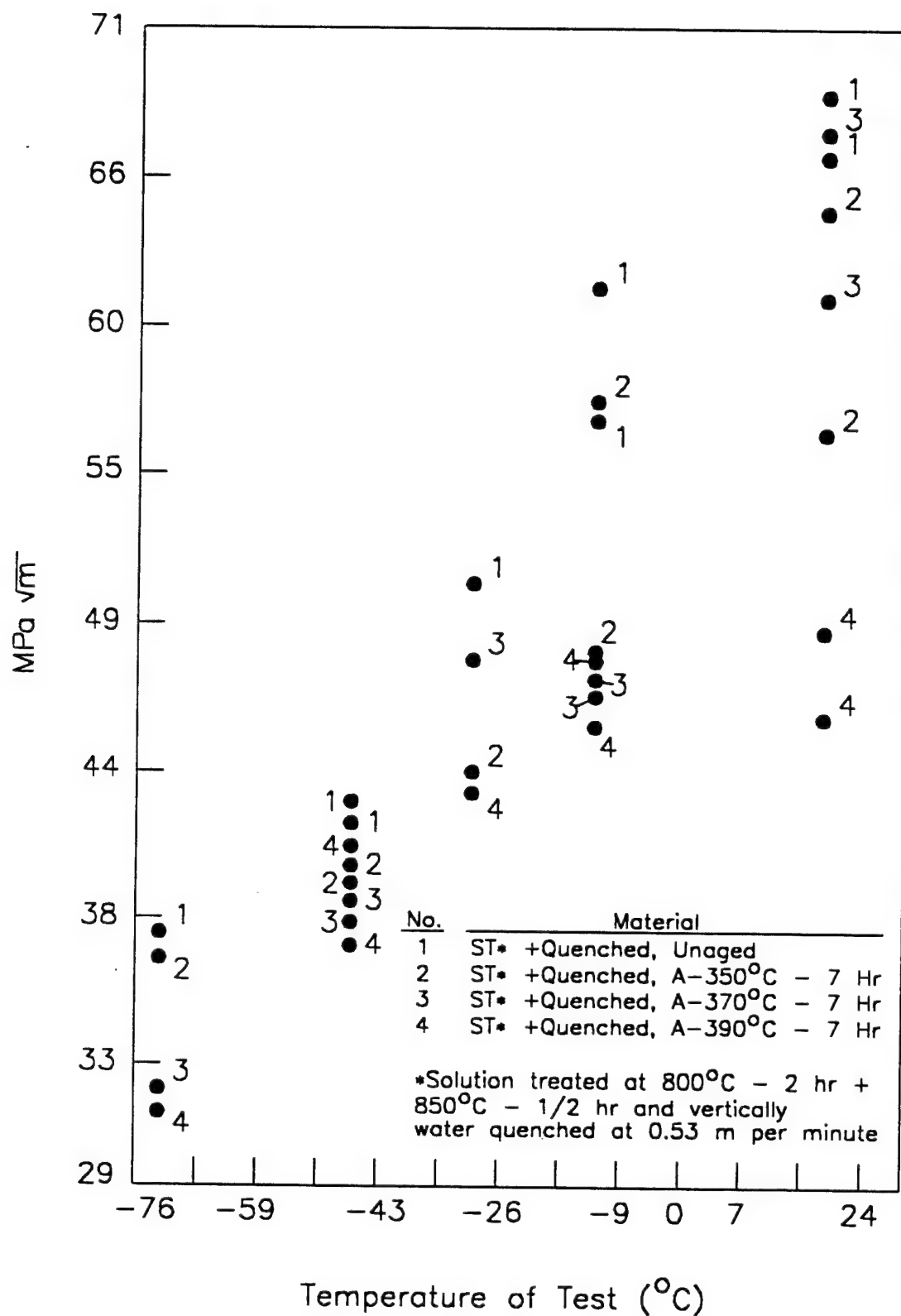


Figure 8. Fracture toughness of aged U-0.75 wt% Ti

Source N bars versus temperature of test.

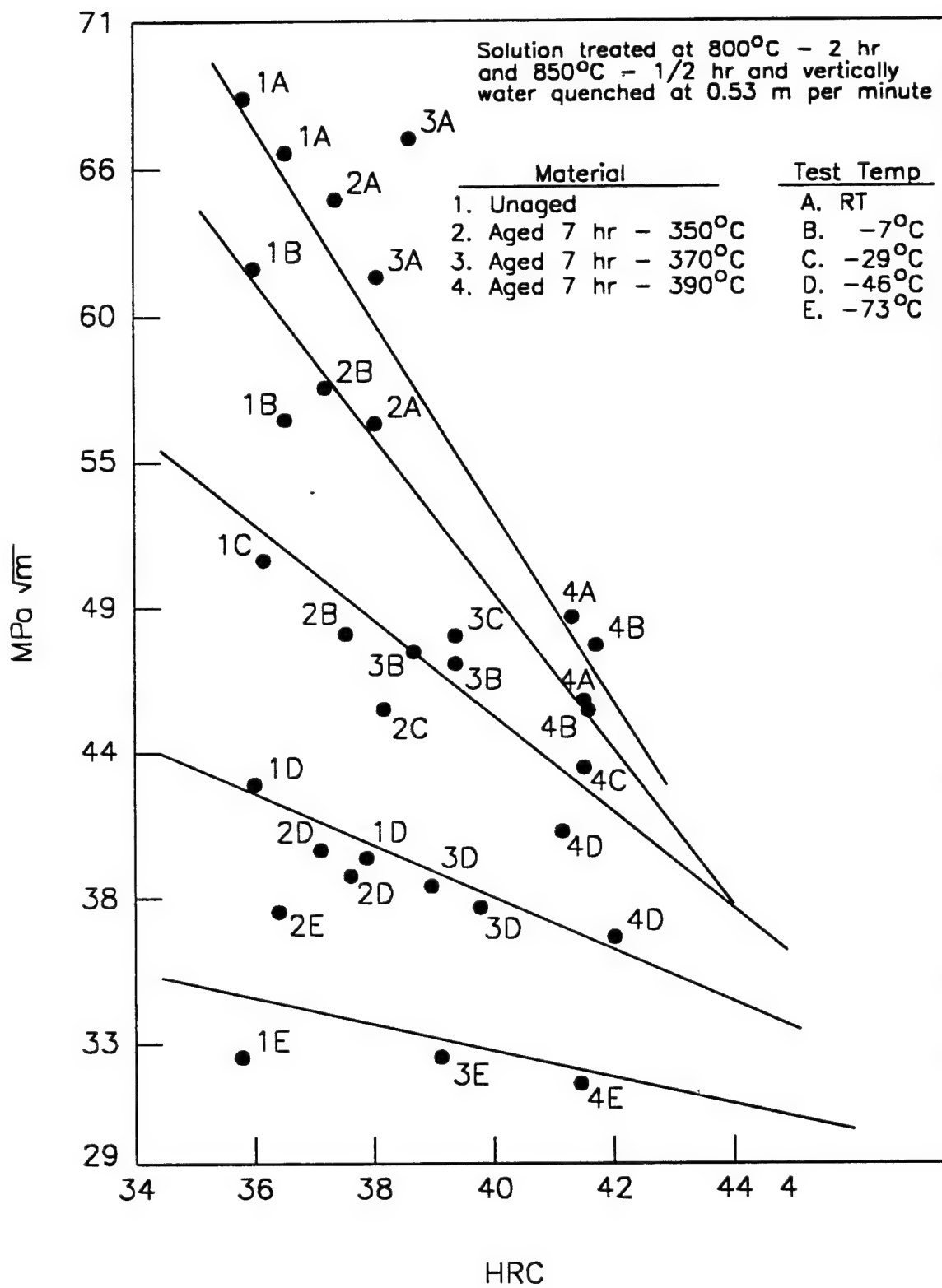


Figure 9. Fracture toughness of aged U-075 wt% Ti
Source N bars (Charpy - K_Q) versus HRC.

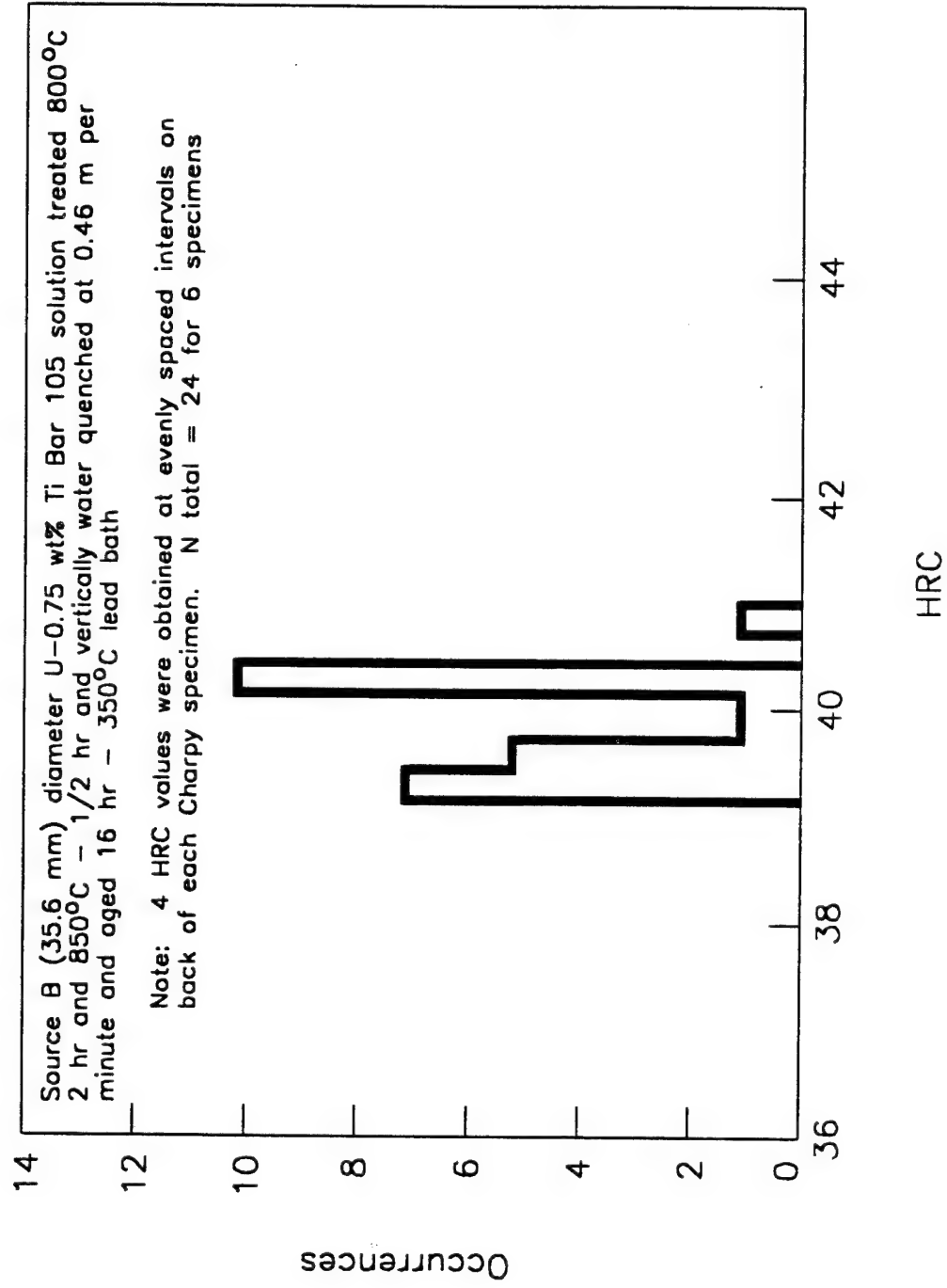


Figure 10. Frequency of Rockwell C readings versus HRC.

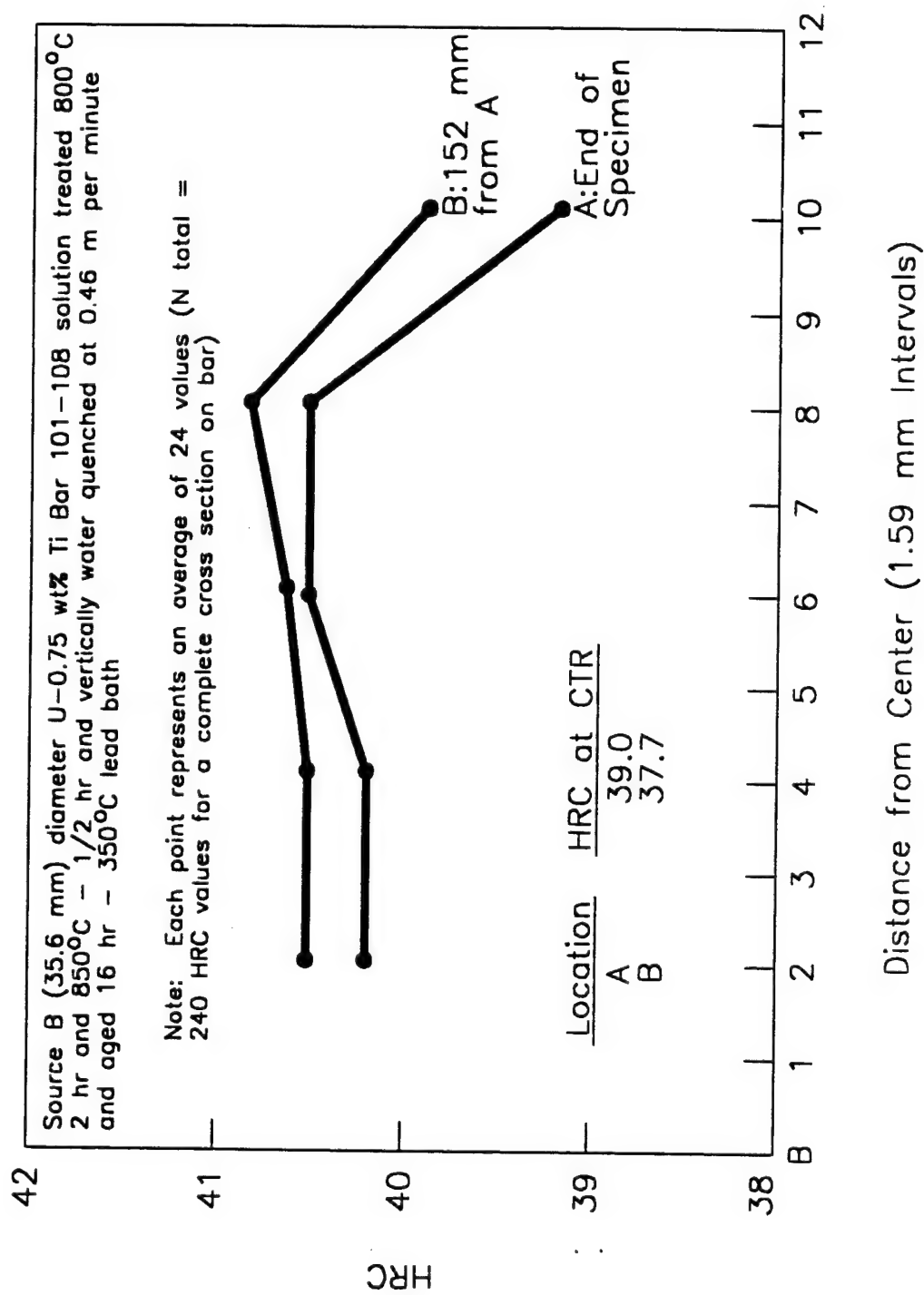


Figure 11. Transverse Rockwell C Hardness versus distance from center.

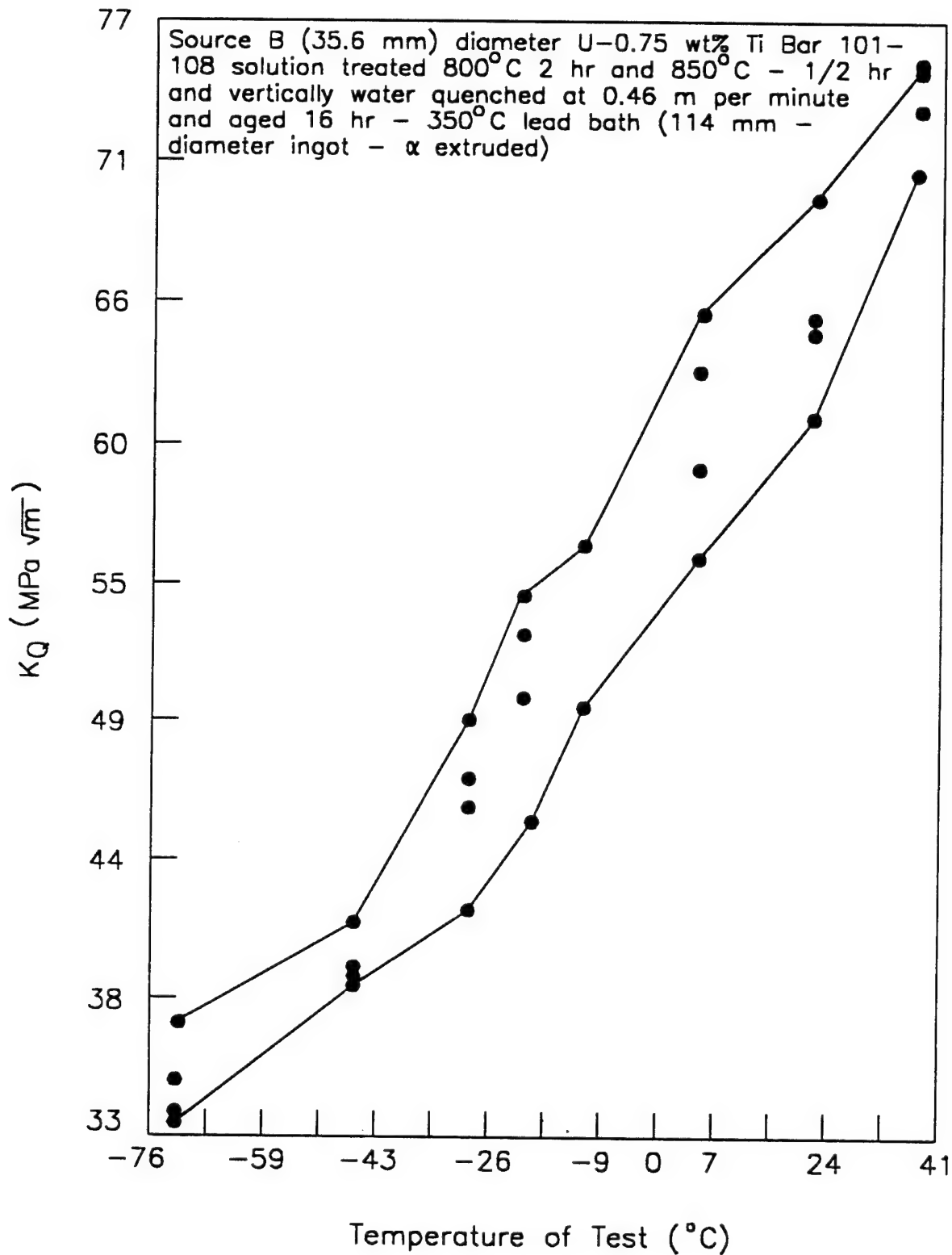


Figure 12. Fracture toughness of aged U-0.75 wt% Ti Source B bars versus temperature of test.

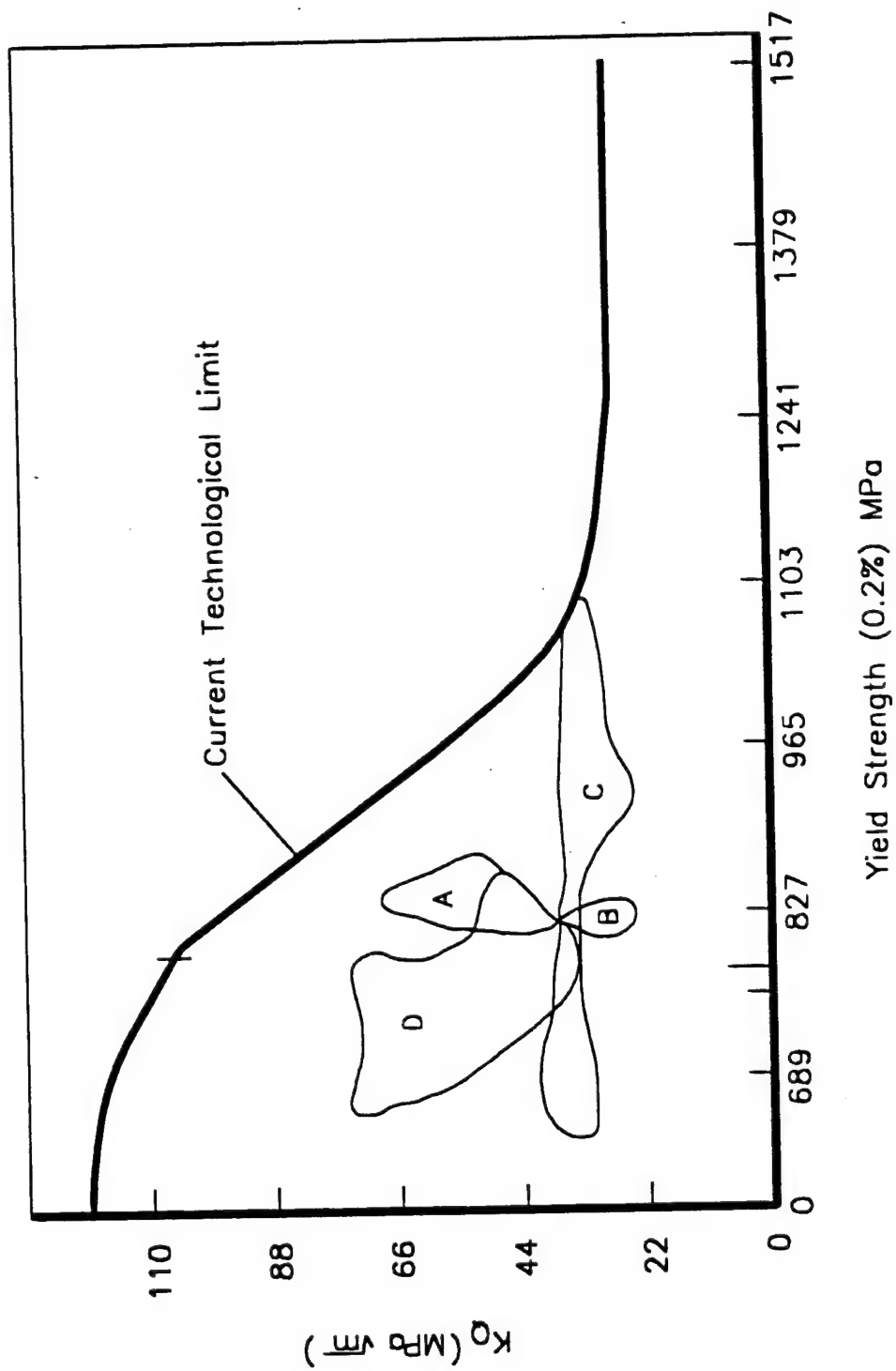
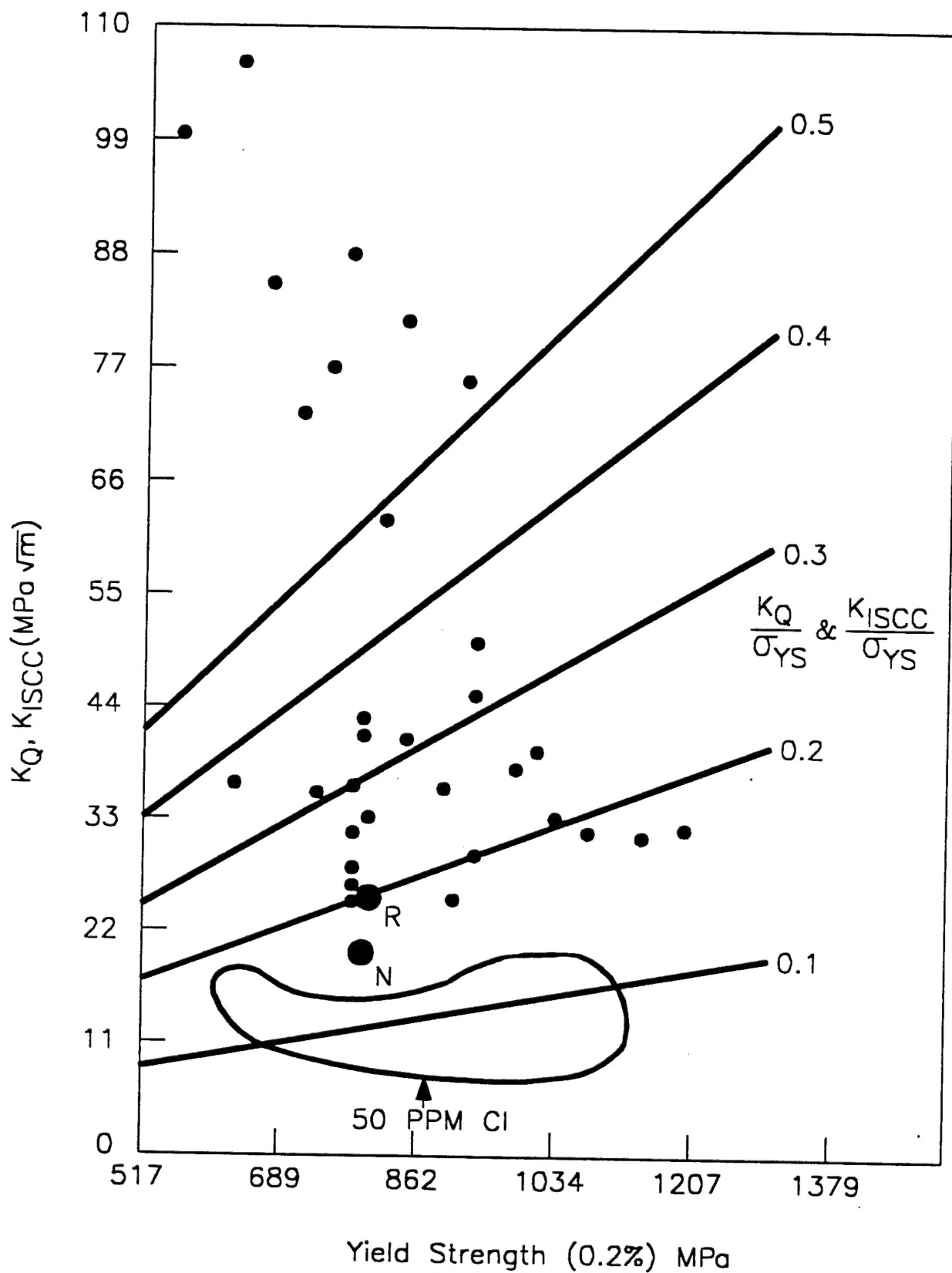


Figure 13. RAD for U-0.75 wt% Ti.



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